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## Permeability After Impact Testing of Composite Laminates

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**Abstract:** Since composite laminates are beginning to be identified for use in reusable launch vehicle propulsion systems, an understanding of their permeance is needed. A foreign object impact event can cause a localized area of permeability (leakage) in a polymer matrix composite and it is the aim of this study to assess a method of quantifying permeability-after-impact results. A simple test apparatus is presented and variables that could affect the measured values of permeability-after-impact were assessed. Once it was determined that valid numbers were being measured, a fiber/resin system was impacted at various impact levels and the resulting permeability measured, first with a leak check solution (qualitative) then using the new apparatus (quantitative). The results showed that as the impact level increased, so did the measured leakage. As the pressure to the specimen was increased, the leak rate was seen to increase in a non-linear fashion for almost all of the specimens tested.

**Keywords:** composites, impact, permeance, leak rate

As composite laminates are being considered for use in liquid propulsion systems, microcracking due to foreign object impact damage becomes very important, especially if a tank or feedline is to be unlined. If a component that carries liquid or gaseous hydrogen develops an area of microcracking, hydrogen can leak out of the component and pose a serious threat to the vehicle. Since it has been shown in the past that a non-visible impact damage can cause a composite feedline to leak [1], a better understanding of the material's resistance to microcracking is needed. Microcracking can also occur due to thermal and mechanical stresses and fatigue, however this study will deal only with foreign object impact damage, a very real threat to all composite parts. The most quantifiable way of determining how much leakage may occur after an impact event is to test the material for permeability. Permeability testing has been used in the past for composites to determine the porosity of rocket nozzle material [2]. Fluid permeability has been tested on some composite structures to be used as fuel tanks [3,4]. Standard Test Method for Determining Gas Permeability Characteristics of Plastic Film and Sheeting (ASTM standard D 1434) exists for gas permeability testing of plastic film and sheeting and it is from this test methodology that the one in this study was adapted.

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For this study, prepreg composed of IM7 fiber in a 5-harness weave architecture impregnated with Bryte Technologies EX1522 epoxy resin was used to construct 4-ply laminates with the lay-up sequence (0/90,0/90)<sub>s</sub>. These specimens were representative of one of the carbon/epoxy systems being evaluated for use in constructing feedlines for future launch vehicles. A drop weight impact tester with a 6.4 mm diameter tup was used to impart varying levels of damage from almost non-detectable to near penetration. The specimens were then secured in a fixture that could supply a positive pressure of Helium gas on one side and allow a leak detection solution to be applied to the other side. This gave a qualitative assessment of permeability-after-impact. The specimens were then secured in an apparatus that could give quantitative results. Since this testing technology is unique, a great deal of test verification was performed and will be presented in this study.

## **Experimental Procedure**

### *Specimens*

The specimens were manufactured from carbon/epoxy prepreg which was in a 5-harness satin weave form. A 91 X 61 cm panel was laid up in a bidirectional configuration on a flat aluminum tool. This gave the laminate a lay-up sequence of (0/90,0/90)<sub>s</sub>. The panel was then vacuum bagged and autoclave cured according to the manufacturer's recommended cure cycle. The cured laminate was then cut into 7.6 X 7.6 cm specimens. The nominal per ply thickness of the specimens was 0.25 mm.

### *Impact Testing*

The 7.6-cm square specimens were impacted at various levels using a drop weight apparatus. The impactor consisted of a 6.4 mm semi-spherically ended tup that was attached to a dynamic load cell to gather instrumented impact data, should it be needed for future analysis. (None of the instrumented data were used in the study presented in this paper). The falling mass had a total weight of 0.88 kg and drop heights of 10.2, 15.2, 20.3, 25.4 and 30.5 cm were used. The specimen was simply supported over a 5.1-cm square opening. Two specimens were impacted from each drop height to give a total of 10 impacted specimens in order to check for repeatability of results. After each specimen was impacted, images of both the front and back surface damage were recorded with a digital camera at a magnification of approximately 5X.

### *Leak Check*

After all specimens had been impacted and the surface damage recorded, the specimens were mounted in an apparatus to check for leakage of helium gas when a positive pressure was applied to one side. A "bubble-type" leak detector solution was used and a digital image was made of the leaks for each specimen. A schematic of the apparatus used to check for leaks is shown in Figure 1.

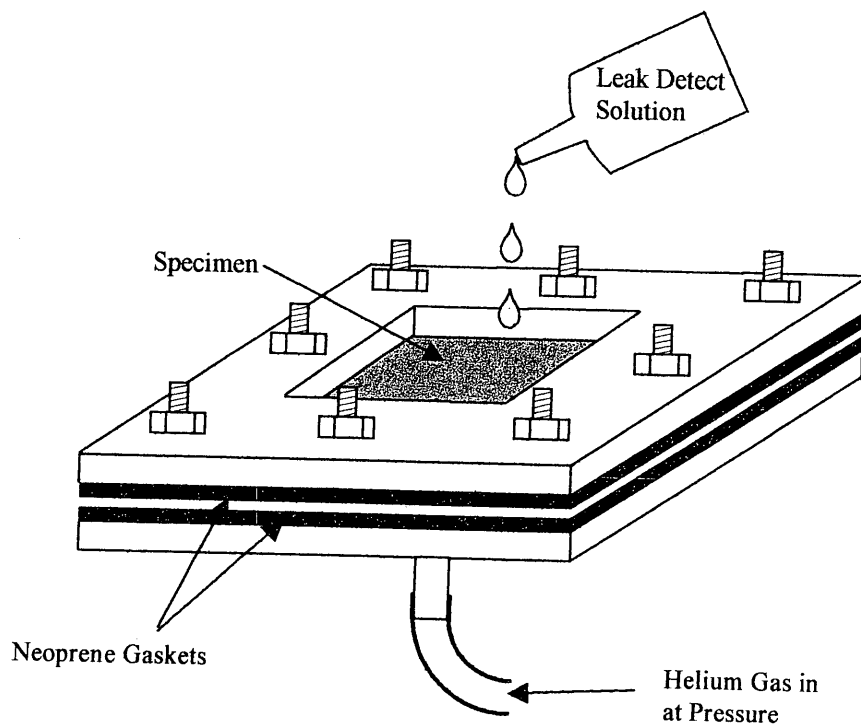


Figure 1— *Schematic of leak detection apparatus.*

A sample image of a leak is given in figure 2.

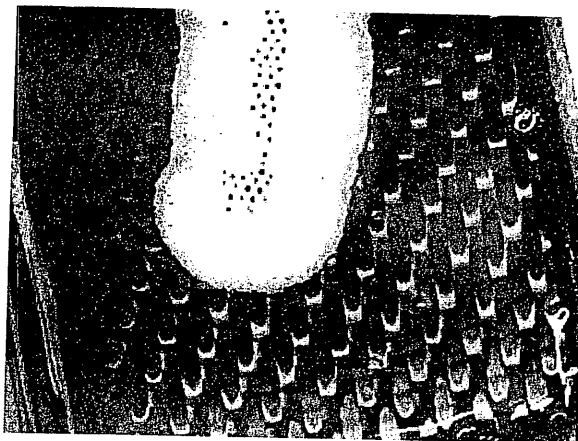


Figure 2— *Specimen displaying a leak using a bubble-type leak detection fluid.*

## *Permeability Testing*

After the specimens had thoroughly dried from the leak detection procedure, they were ready for permeability testing. The following six sections will be devoted to a discussion of the apparatus used and its limitations and verification.

*Permeability Apparatus*—The apparatus used in this study is based loosely on ASTM Standard D1434 using the Volumetric Technique. This apparatus could have been used, but modifications would have to be made for the larger specimen size needed (to include all of the impact damage) and a much more simple apparatus could be used since relatively high rates of permeance were being measured. The apparatus basically consisted of a specimen holder that isolated the two surfaces of the composite laminate and allowed a positive pressure of gas to be applied to one side and the gas that leaked through the specimen to escape out of the other side. The escaping gas rate was measured via a volumetric displacement method using a liquid “slug” in glass tubes of various diameters. The rate of volume change was measured with a stopwatch so the amount of gas escaping through the specimen could be calculated in volume per unit time.

It should be noted at this point that, strictly speaking, the term “permeability” can only apply to homogeneous materials and is defined as the “permeance” times the specimen thickness. Permeance is defined as the transmission rate per applied pressure of gas, which is what is being measured in this paper. However, the term “permeability” has been used in the composites industry much more than “permeance” to indicate a transmission rate and it will be used in this paper in order to avoid confusion.

A sketch of the apparatus is given in figure 3 to aid in the discussion that follows. A test or “run” was typically conducted in the following manner.

The 7.6 X 7.6 cm square composite specimen was centered and sandwiched between the top and bottom aluminum plates. A small amount of vacuum grease was placed around the edges of the specimen to ensure a good seal between the specimen and the neoprene gaskets. The eight bolts and nuts were then used to secure the specimen between the two plates. Each bolt had 1130 N•m of torque applied. The specimen holder was then secured to a cantilever beam above a laboratory table with a c-clamp. An inlet hose from a helium supply tank was then inserted over the bottom nipple to supply the positive pressure to the specimen. A precision pressure gauge was included in the supply line so an accurate reading of the amount of pressure being applied to one side of the specimen could be determined. Typical values of applied pressure ranged from 20.7 to 344 kPa. A liquid “slug” or “blank” was then introduced into the glass tube. Alcohol was usually used as the liquid and enough to introduce a slug length of approximately 7 to 20 cm was inserted into the top of the tube. The alcohol was then allowed to fall into the bottom U-shape of the Tygon® tubing that was snugly attached to the glass tube. A period of about ten minutes was allowed for all of the liquid to flow down the glass tube and for the specimen to be subjected to the test conditions. The loose end of the tubing joined to the glass tube (the other end of the U-shape) was then attached to the upper nipple of the specimen holder to channel all leaking gas into the tubing and force the slug up the glass. The slug was allowed to reach steady state (constant velocity) and then the time needed to travel a given distance was recorded. This procedure was repeated for various applied pressures of helium so a pressure versus flow rate chart could be created.

The next section will present equations and experimental results to show how various parameters could affect the flow rate data.

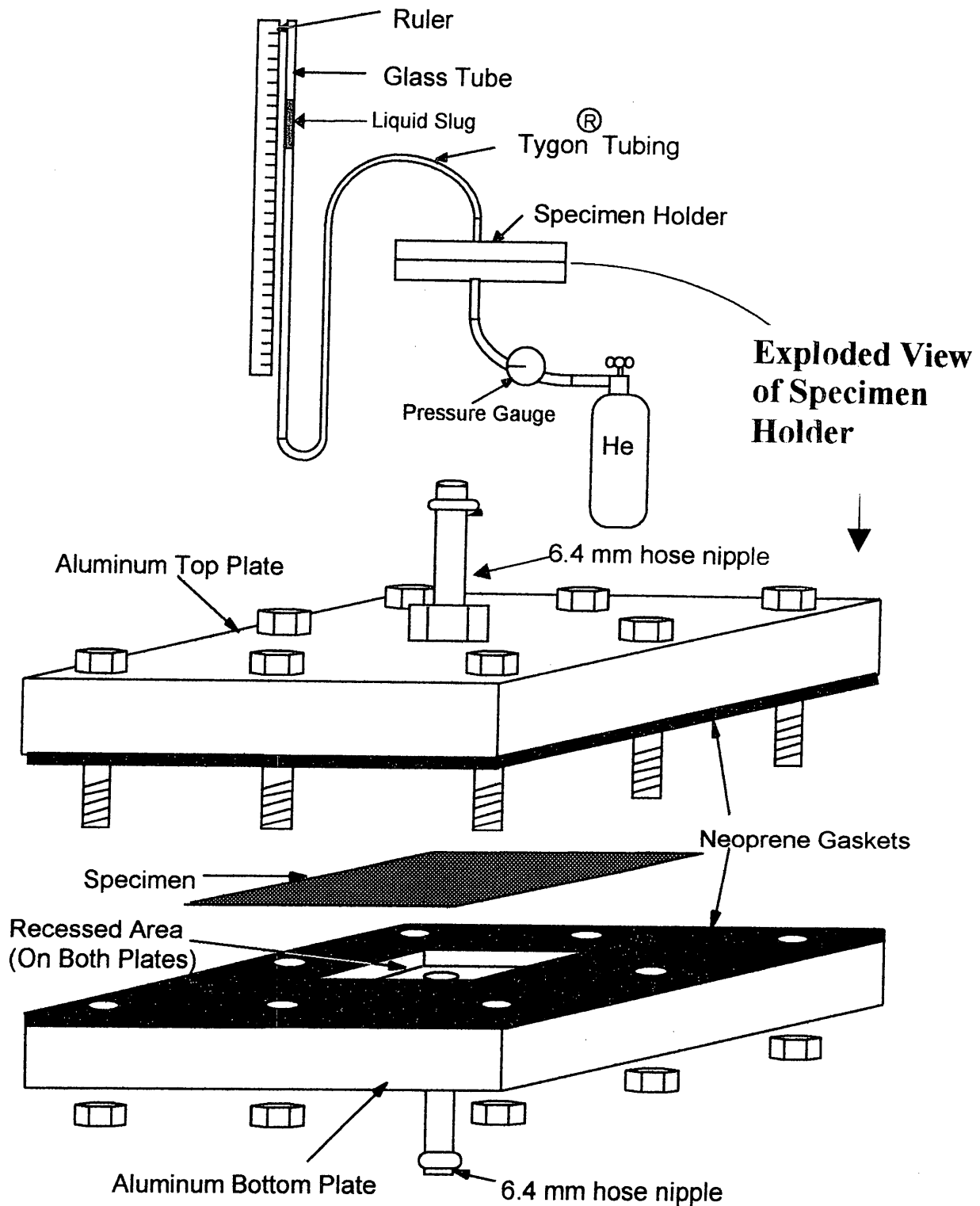


Figure 3—Sketch of permeability apparatus.

*Analysis of Apparatus*—From past experience and intuitive deductions, it was determined that certain key variables in performing the test may affect the outcome of the flow rate data. These variables were examined using equations of laminar flow to see what variables would be most likely to affect the data. Figure 4 will be useful in the following discussions.

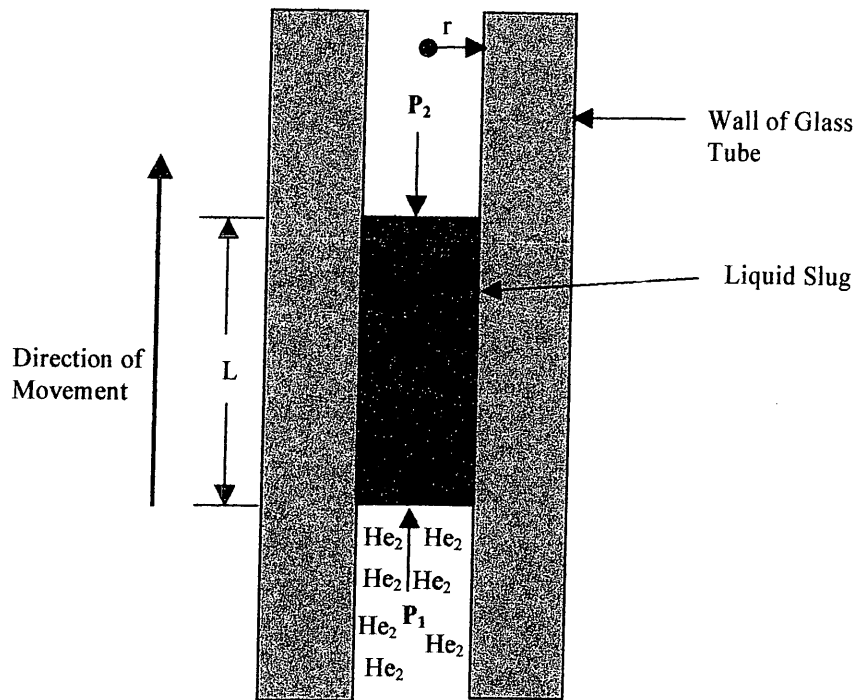


Figure 4—Schematic of liquid slug in glass tube.

Since the end of the tube is unrestricted,  $p_2$  will always equal atmospheric pressure  $p_a$ . At the beginning of the experiment, the volume between the top of the specimen and the slug is also at atmospheric pressure (i.e.  $p_1$  also equals  $p_a$ ). Thus the additional force needed to keep the slug in the tube from falling back down is given by:

$$F = \pi r^2 L \rho g \quad (1)$$

where

$r$  = Inner Radius of Glass Tube

$L$  = Length of Liquid Slug

$\rho$  = Density of Slug Liquid

$g$  = Acceleration due to Gravity

Thus the additional pressure needed is  $F/\text{Area} = F/\pi r^2$ , or:

$$p_g = L\rho g \quad (2)$$

This equation indicates that the pressure needed to keep the slug from falling back down the tube is proportional to the slug length and the liquid's density. Thus to minimize this term, a short slug length and a low-density liquid are desired. It should be noted that if the tube is slanted toward the horizontal position, less of the slug liquid will be acting in a downward manner and L will be lessened by the amount  $\sin\theta$  where  $\theta$  is the angle the glass tube makes with the horizontal.

Assuming the slug to be alcohol, the pressure in Pascals needed to keep the slug from falling back down is given by;

$$p_g = 7742(L) \quad (3)$$

where L is slug length in meters. Thus assuming a 0.1 m slug length,  $p_g$  will be on the order of 774.2 Pa which is less than one percent of atmospheric pressure.

Once the slug is moving at a constant velocity up the tube (steady state), the ~~viscous forces of the liquid need to be overcome. This force is given by:~~

$$(p_1 - p_2) = \frac{dV}{dt} \frac{8\eta L}{\pi r^4} \quad F = 8\pi\eta Lv \quad \text{Poiseuille's law states} \quad (4)$$

Where;

$\eta$  = Viscosity of Liquid used for Slug

L = Length of Slug

~~$v$  = Velocity of Slug Traveling up the Glass Tube~~

$r$  = radius of tube

$dV/dt$  = Change in volume per unit time

Thus the ~~additional~~ pressure needed to overcome the viscous forces of the slug is  $\frac{8\eta Lv}{r^2}$ , needed to overcome the viscous forces of the liquid as well as the additional  $p_g$ .

$$p_\eta = \frac{8\eta Lv}{r^2}$$

The volume rate  $\frac{dV}{dt}$  can be given as  $\pi r^2 v$  where  $v$  = velocity of the slug traveling up the glass tube

This equation indicates that the pressure needed to overcome the viscous forces of the slug are proportional to the liquid's viscosity, slug length, velocity and inversely proportional to the square of the radius of the tube. Thus to minimize this term, a low viscosity liquid, a short slug length, a low slug velocity and especially a large tube radius are all desired. Assuming a slug length of 0.1 m, a tube radius of 0.0015 m, a velocity of 0.01 m/sec and the slug liquid as alcohol, the pressure required to overcome the viscous forces is about 430 Pascal or about one-half of one percent of atmospheric pressure.

As a check for possible errors introduced by these forces, some experiments were carried out varying the angle of the tube, the liquid in the tube, the length of the liquid slug and the tube size. The results of these experiments follow.

*Vertical versus Slanted Glass Tube*—It was determined early on that if the glass tube could be laid down at a slant, rather than being vertical, the testing would be much

simpler. Thus a series of tests were performed with the glass tube in a vertical position and the glass tube laid down at an angle of 5 degrees. Specimen 6B was used for all of the tests and alcohol was used as the liquid for the slug. Excellent repetition of data was obtained and typical results from a vertical versus a slant tube are shown in figure 5.

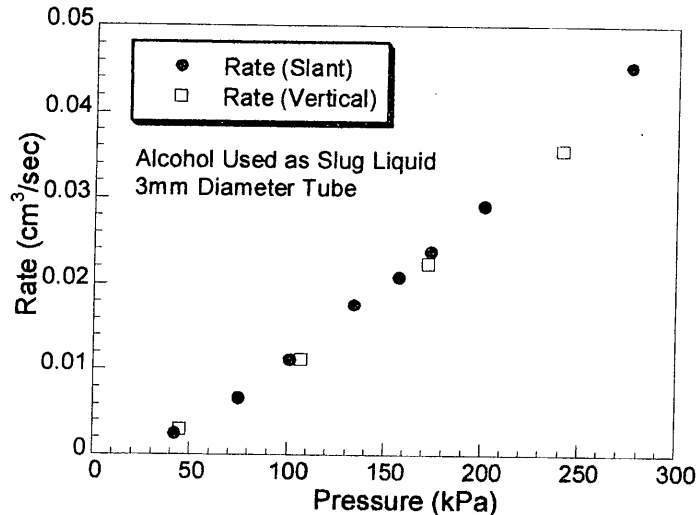


Figure 5— Flow rate results of vertical and "slanted" glass tube.

Since no differences were found, all subsequent tests were performed with the glass tube placed on a wood board angled at 5 degrees.

*Type of Liquid Used as a Slug*—Alcohol was typically used as the liquid for the slug, but the use of water and alcohol with PhotoFlo® to reduce surface tension was examined. Again, specimen 6B was used for these tests and the blank length kept at approximately 3 inches. Results are shown in figure 6.

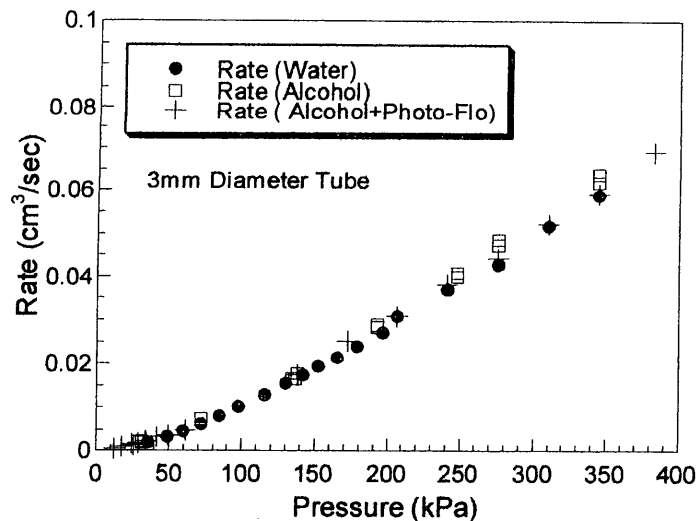


Figure 6— Flow rates for Specimen 6B using various liquids for slugs.

Since no difference was found for the liquid used as the slug, alcohol was chosen for subsequent testing due to its ease of cleaning and fast evaporation rate.



*Length of Slug*—It is very difficult to obtain a consistent slug length when placing the liquid in the glass tube. Thus a series of tests were performed to see if the slug length affected the measured flow rate. Specimen 5A was used for these data. The results are shown in figure 7.

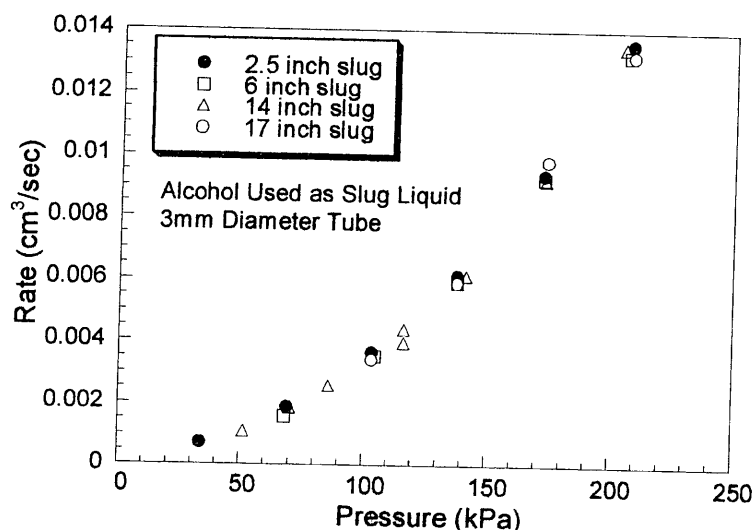


Figure 7—Flow rate using different lengths of liquid slugs for specimen 5A.

No differences in measured rates were noted so slug length was not a factor and any convenient length could be used.

*Size of Glass Tube*—Three different sizes of glass tubes were used having inner diameters of 0.4, 1.2 and 3.0 mm. For a given flow rate, the slug will obviously travel slower in the larger diameter tube and more rapidly in the smaller diameter tube. Since the location of the slug at various times needs to be measured, the slug cannot be traveling too fast, or accurate readings cannot be achieved. Also, if the flow rate is small, it can take a very long time to get a reading, unnecessarily slowing the data acquisition process. To examine the affect of tube size on flow rate, three tube sizes were used to measure the flow rate on specimen 5A. The results are presented in figure 8.

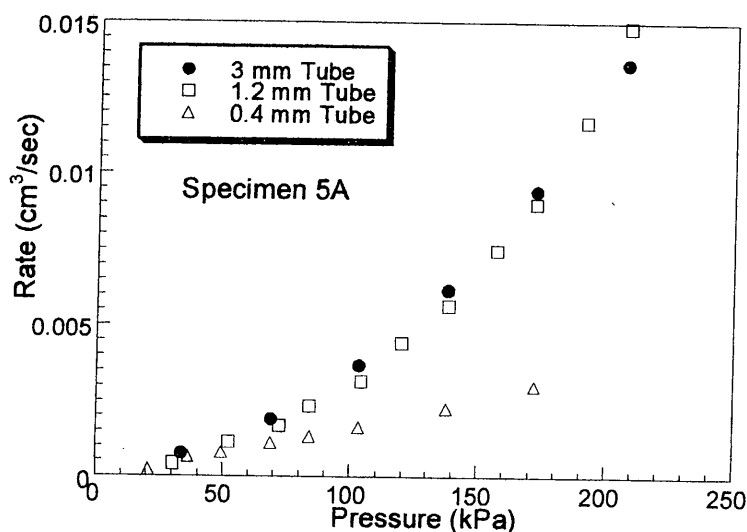


Figure 8—Flow rate measured on specimen 5A using different size tubes.

Since a difference is clearly noted for the 0.4-mm tube, this test was performed on specimen 6B. The data are given in figure 9.

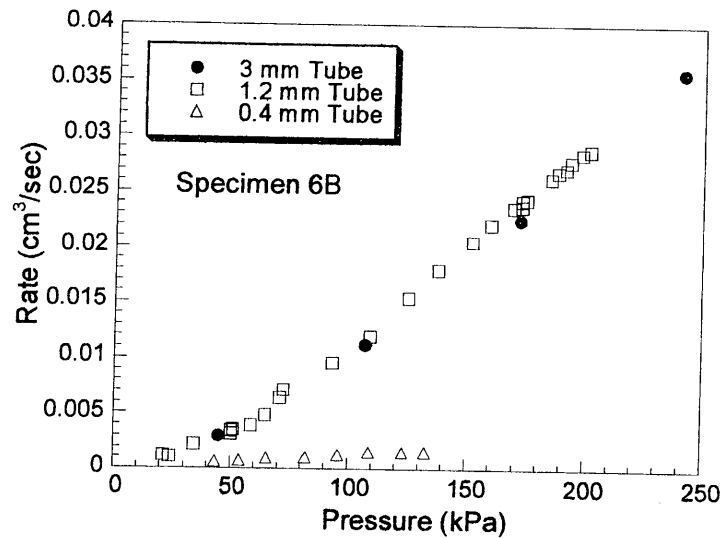


Figure 9— Flow rate measured on specimen 6B using different size tubes.

Again the data shows a much lower measured flow rate for the 0.4 mm tube whereas the 3.0 mm and 1.2 mm tubes give excellent agreement

Using actual data, the pressure needed to overcome the viscous forces of the slug can be found from equation (5). The results for the three tube sizes used are shown graphically in figure 10 as a function of flow rate  $v$ . A slug length of .01 meters was assumed. The liquid was assumed to be isopropyl alcohol at room temperature.

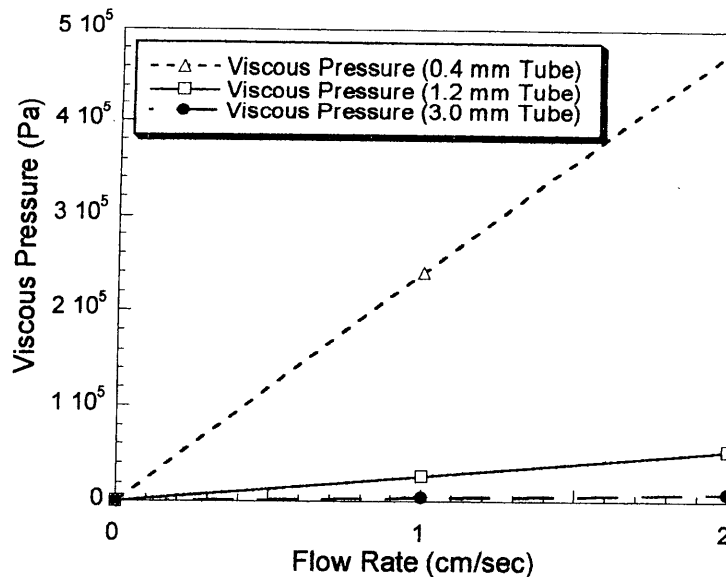


Figure 10— Plot of viscous pressure on slug as a function of flow rate.

This plot shows just how critical the size of the tube can be when it comes to viscous forces. Not only does the smaller tube give rise to much higher viscous forces for a given flow rate, but for a given specimen, the flow rate will be highest in the smallest tube, further increasing the viscous forces that need to be overcome.

## Results

This section will present results of the impact testing, the leak check testing and the permeability testing. Results will be limited to the three lowest impact energy levels used since higher level impacts produce very visible damage with large leak rates as expected.

### *Impact Testing*

A total of 10 specimens were impacted, two at each of five energy levels. The resulting visual surface damage for the 3 lowest level energy impacts are presented in figure 15. Duplicates are not presented since the visual damage was nearly identical in every case.

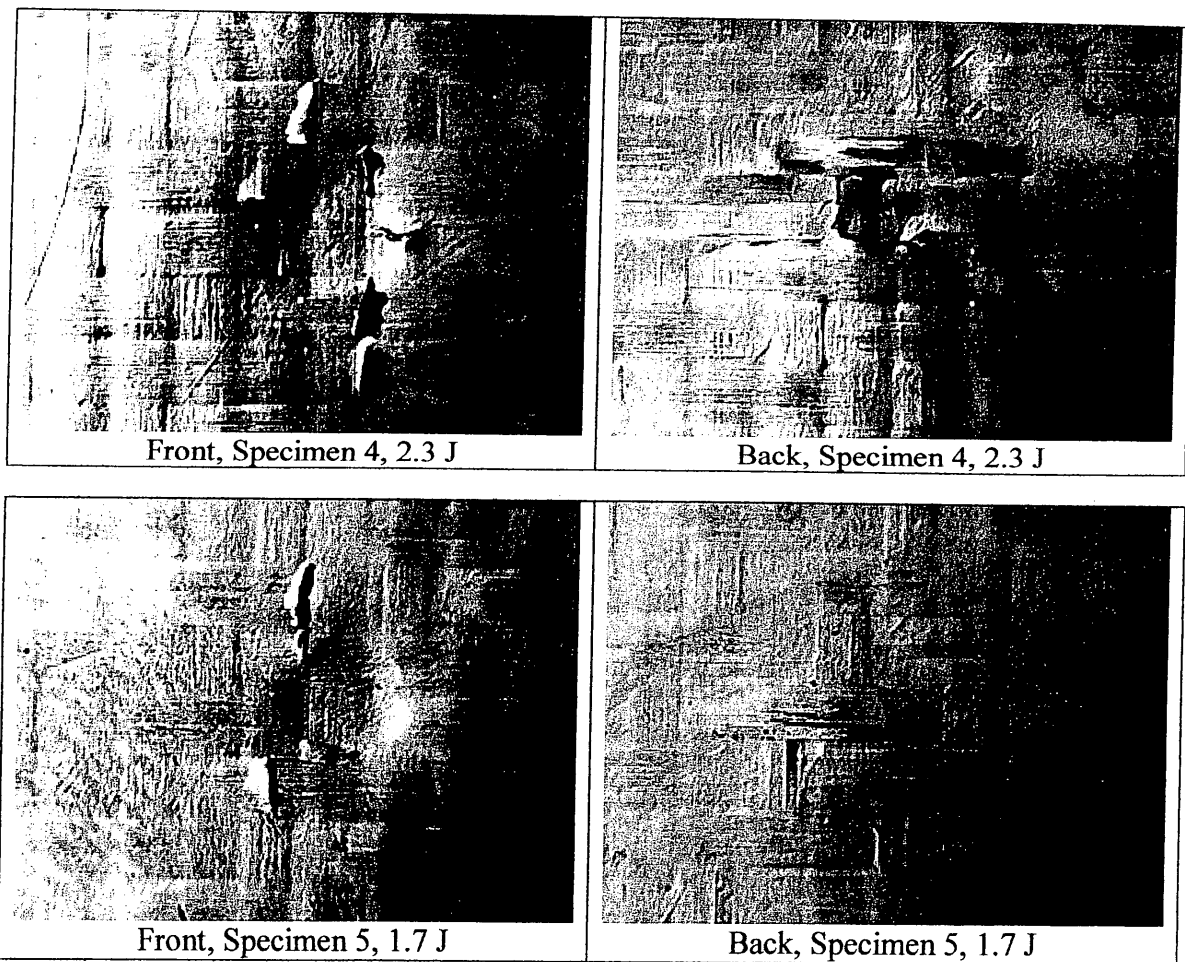


Figure 15—*Surface Views of Impacted Specimens*

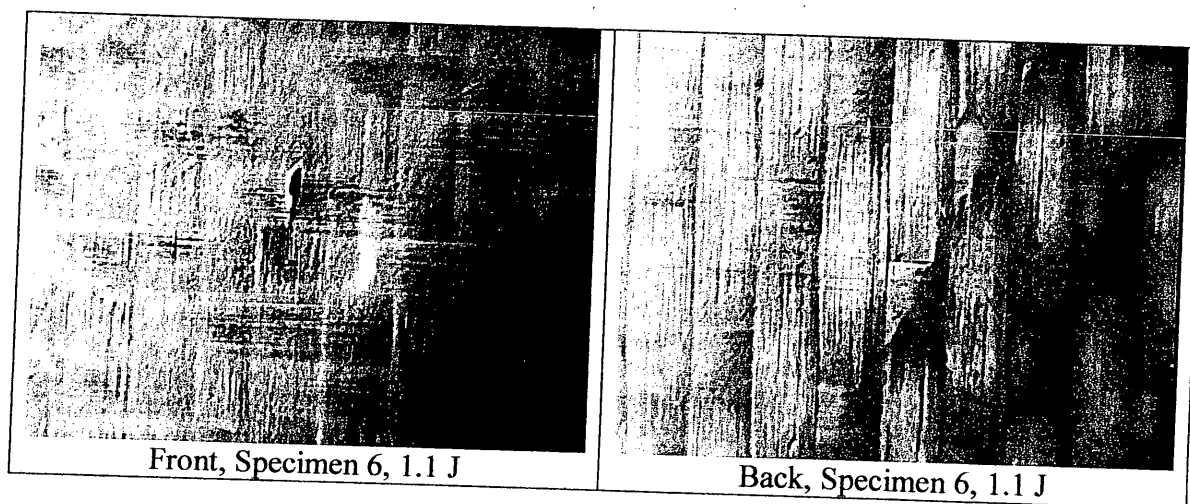


Figure 15— *Surface views of Impacted Specimens (continued).*

Damage of some form can be noted on all specimens, even those impacted at the smallest impact energy of 1.1 J.

#### *Leak Check Testing*

Figure 16 shows photographs of the specimens when subjected to a pressure of 10 PSI on one side and a bubble type leak detection solution placed on the other. Results from all specimens are included since these results did not show the duplication that was seen in the visual results. The impact energy level is included below each image.

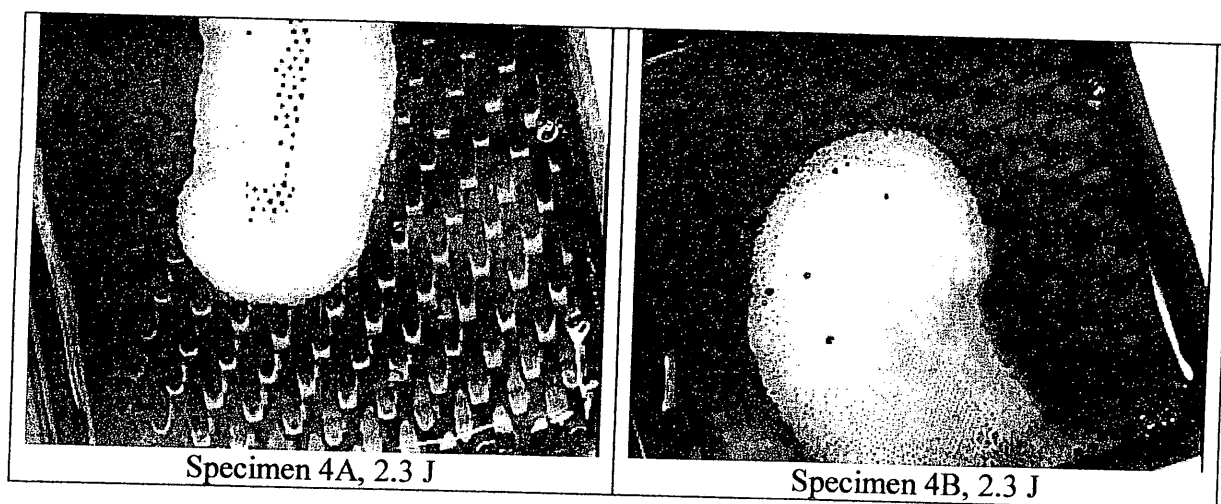


Figure 16— *Leak Check Images.*

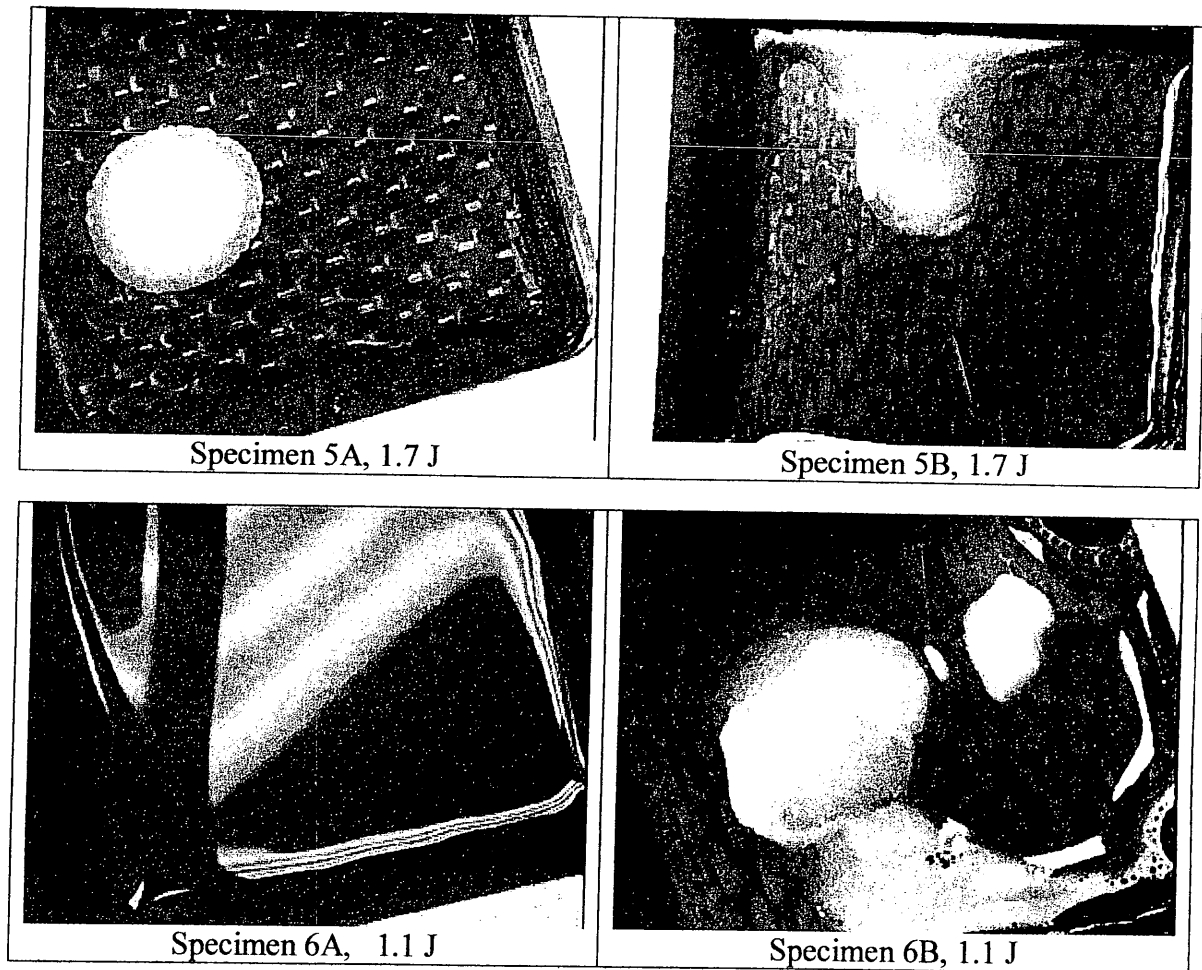


Figure 16—*Leak Check Images (continued).*

All samples, with the exception of 6A, showed leakage. The larger the bubbles are in the pictures, the higher the leak rate. As the impact damage becomes less severe, the bubbles become smaller. In fact, in specimens 5A and 6B, the leak rate is such that the leak detection fluid forms a fine “foam” that emanates from the impacted area.

#### *Permeability Testing*

Figure 17 shows plots of flow rate versus pressure for the samples tested. Specimen 6A showed no permeability (as would be expected from figure 16).

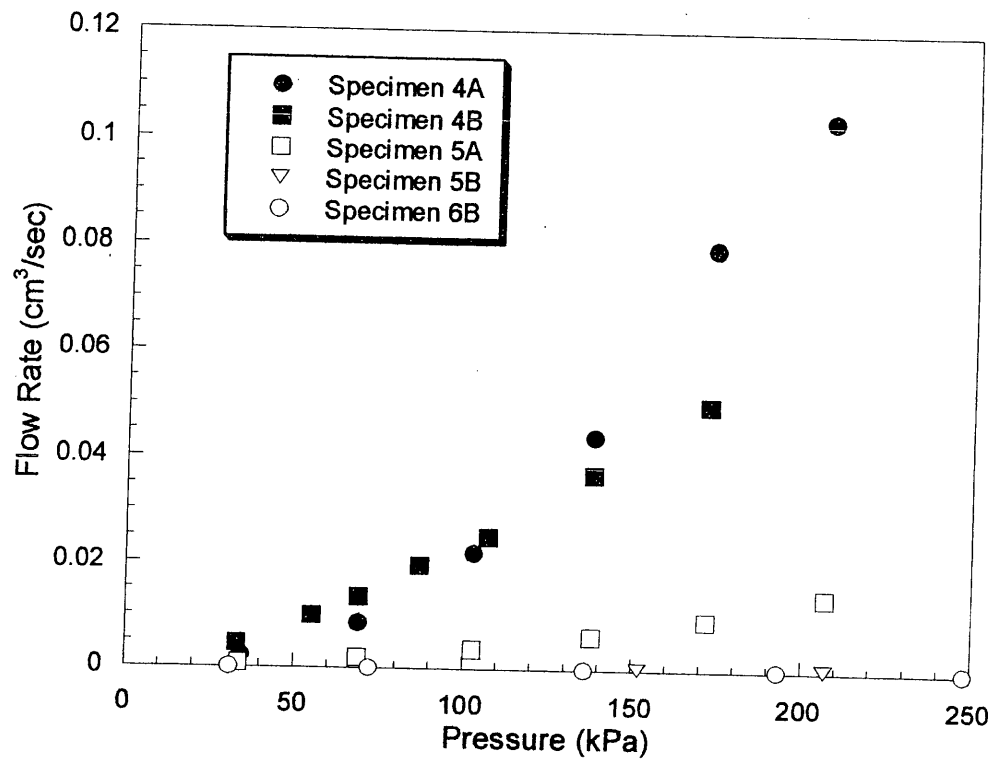


Figure 17—Flow rate (permeability) versus applied pressure for all specimens tested.

Figure 18 shows only the specimens with the two lowest leak rates for more clarity.

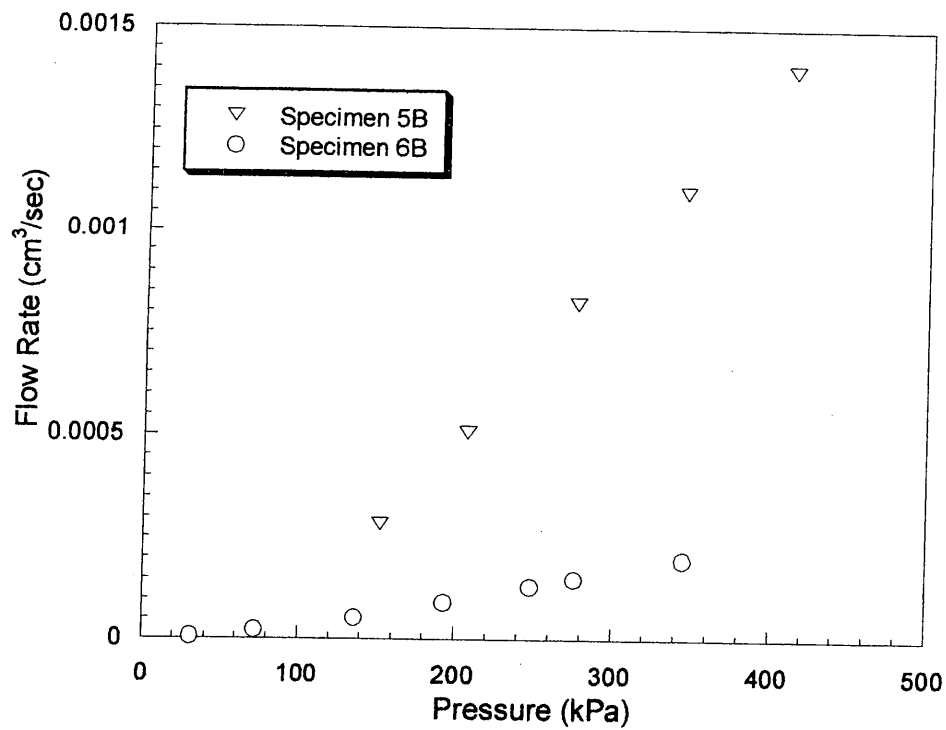


Figure 18—Flow rate (permeability) versus applied pressure for two lowest leak rate specimens tested.

In general, the larger the impact energy, the higher the flow rate for a given applied pressure, which is expected. The amount of non-linearity in flow rate versus applied pressure also varied between samples, however most of the non-linearity that is observed is small.

## Conclusions

A simple test apparatus can be used to measure permeability of impact damaged composite specimens, as long as the flow rate through the sample is not too great. Using the apparatus presented in this study, it was demonstrated that tube size is critical in obtaining proper flow rate readings. Too small of a tube can give rise unacceptable levels of viscous forces on the liquid "slug" inside of the tube, resulting in flow rate measurements that are too small. The type of liquid, length of the slug and angle that the tube makes with the horizontal were all shown to have little or no affect on the flow rate readings.

For permeability-after-impact testing, the residual flow rate usually has a non-linear dependence on the applied pressure, increasing more rapidly as a higher pressure is applied. Thus the permeability cannot be stated as a constant per unit of applied pressure.

The four-ply laminates tested in this study showed leakage after impact, even when visible damage could only be detected with magnifying techniques.

The qualitative measurement of leakage with the bubble-type leak detector solution corresponded with the qualitative permeability measurements.

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